

The Development of a Standard for Contact Transient Methods of Measurement of Thermophysical Properties

R. P. Tye ¹, L. Kubicar ² and N. Lockmuller ¹

1. National Physical Laboratory, Teddington, United Kingdom
2. Slovak Academy of Sciences, Bratislava, Slovakia

ABSTRACT

Contact transient methods, some of which are available as commercial forms, are now widely used worldwide for thermal properties measurements on broad ranges of materials used in physical, chemical and medical applications. However, in many cases the claimed measurement uncertainty has not been substantiated while in others – especially for the multi-property techniques – internal inconsistencies in measured and/or derived values are clearly apparent.

Following recommendations of participants of two workshops held on the subject in Wurzburg (1999) and Cambridge MA (2001) NPL agreed to coordinate a task to develop a standard test-method for these techniques. This involved using inputs provided by a small group of individuals from organizations in several European countries and also taking note of comments from other interested parties via the internet during the course of the development.

Details are provided on the resulting document, which takes the form of a generic standard containing appropriate details and related information common to all techniques. These sections include the scope, theory, summaries of method, basic apparatus and experiment, the influencing factors, specimen requirements, procedure and recommended approach to analysis of the experiment and calculation of the results. In addition there are six Annexes, each of which contains additional information that applies to a specific technique. Finally, the document proposes a recommended approach to verification of a technique together with a list of appropriate reference materials having known values for one or more properties. The status of intercomparison studies will also be reported.

BACKGROUND

Heat transmission through materials is traditionally characterised by the basic partial differential equation for heat transport (heat equation) based on the Fourier law. This theory of the heat transport was developed for homogenous materials and provides three parameters: thermal conductivity, specific heat and thermal diffusivity. All three can be measured by individual techniques and one test of validity (data consistency relation) for dense homogenous specimens is the relation between the measured parameters where thermal conductivity is the product of thermal diffusivity, specific heat and density.

When no structural transformation exists the physics behind these parameters is connected with phonons and electrons. Standard methods of measurement have been available for thermal conductivity, thermal diffusivity and specific heat. In general these have been based on so-called steady state or equilibrium techniques which usually involve various large sizes or amounts of material as specimens and require especially long measurement times.

While most traditional materials have polycrystalline or amorphous structures many of the more recently developed materials possess a combination of both (composites, layered structures etc), have a porous or multiphase structure or are in very limited forms. From the physical point of view such materials represent structures that are on the one side in an equilibrium state and on the other side in a highly non-equilibrium (metastable) state. Measurement techniques used for characterisation of material property are adjusted predominantly just for traditional materials and the current parameters have been suitable for such characterisation.

The use of such materials has produced a broad spectrum of new issues and problems. The parameters used up to now do not necessarily represent the properties required to be measured while the models used do not represent the processes being studied. This is a challenge for the physicist to build models of these sophisticated, highly inhomogeneous materials that should provide a minimum of reliable parameters. The development of measuring techniques is going hand in hand with the efforts to verify the constructed models. In a majority of cases the thermal properties – thermal conductivity or diffusivity, heat capacity and emissivity and/or absorption – are used up to now. However, the physics behind these parameters for the newer materials and their application is now an open question. Thus, the use of traditional parameters for the newer materials needs to be redefined and conditions for their application in practice need to be found.

The sheer volume of current materials and their applications combined with the fact that their availability in limited sizes and forms only make steady state methods unsuitable for

measurement requirements. There has been a growing need for the development of new methods which are more rapid, use smaller specimens and are multi-property in concept.

As a result much attention has been paid to the development of the so-called transient contact methods (TCM) such that several commercial forms are already available and widely used. Table 1 summarises the major forms of the methods which are all based on a common principle. Additional variations are also in development.

Table 1. Summary of basic forms of TCM				
Name of method	Heat source geometry	Way of heat production	Heat source/temperature sensor configuration	Measured and/or derived parameter
Hotwire/probe/strip	line, strip	step-wise	united ¹ or separated ²	λ , a, (c & E in some experimental forms)
Pulse Transient	plane	pulse	separated	a, c, λ
Step-wise Transient	plane	step-wise	separated	a, c, λ
Hot Plate Transient	plane	step-wise	united	E
Hot Disc Transient	disc	step-wise	united	a, c, λ
Gustafsson Probe	concentric circles	step-wise	united	a, c, λ

1 = one sensor; 2 = two sensors

λ = thermal conductivity

a = thermal diffusivity

E = thermal effusivity

c = specific heat

Because of their apparent “simplicity” in concept and realisation these techniques have become very attractive and popular. In particular the fact that times of assembly and measurement are reduced from hours to minutes or seconds, enabling many specimens to be evaluated in the same time as one being evaluated by a standard technique, make them ideal for current requirements. One of the major attractions is that some are multi-property in concept. Thus the thermal diffusivity and heat capacity can be measured directly, often simultaneously, and hence thermal conductivity can be determined from the accepted relationship involving these properties and the density, i.e. automatic fulfilment of data consistency. Some more recent versions claim to enable all three properties to be determined directly from different parts of the temperature/time relationship(s). Furthermore, precision claims for these techniques are such that they are judged to be comparable to or better than standard methods. Finally, an important feature is that certain forms lend themselves to be considered as being suitable for use in “on line” applications in manufacturing and processing or in miniaturised form for in situ and in vivo applications.

Due also to the significant advances that have occurred in instrumentation and computer hardware and software it has been possible to automate fully each technique with results provided by an analysis using specialised often proprietary software. Because of this factor measurements are now often undertaken in many cases by operators having little direct experimental and/or materials experience who place reliance on the fact that the methodology realises the solution of the appropriate model exactly and that the software represents this realisation.

As these methods become more widely used, results of work on a number of homogeneous material types are becoming available from publications in fields of science and engineering. Examination of data indicates that the claimed high precision for one or other property by the particular technique (often 3% or better) is not substantiated since results for the same or similar material from two sources can differ by 10% and often more. Furthermore, in some cases results are often found to be internally inconsistent in that the measured thermal conductivity and/or that derived from thermal diffusivity and specific heat can be significantly different from the accepted value. In certain circumstances there can or may be valid reasons such as anisotropy, heat flow direction, convection, radiation etc why some differences in value for thermal conductivity exists. However they occur for materials or circumstances where none should exist and thus the particular method itself becomes of questionable use.

Recent examples to illustrate some particular issues include measurements on Pyroceram 9606 using the hot wire and Gustafsson probe methods.

During the program of work for certification of Pyroceram 9606 thermal properties some parallel wire measurements were undertaken up to 1000 °C on a specimen which was somewhat

smaller than the recommended size. The results for this specimen are shown in Figure 1 which also contains the final certified values for the material. These were obtained using standard guarded hot plate and both resistive and parallel mode hot wire tests on suitably larger specimens.

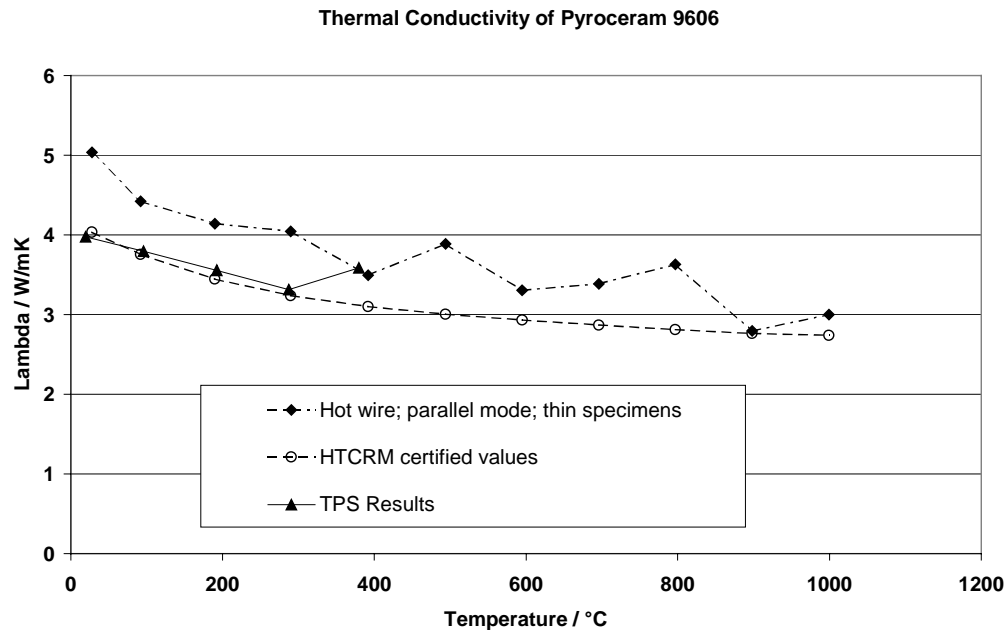


Figure 1. Thermal conductivity of thin pyroceram specimen measured by hot wire method in parallel mode.

It can be seen that the directly measured thermal conductivity is significantly higher than the certified value and outside the expanded uncertainty. Furthermore the derived thermal conductivity obtained from the individually measured thermal diffusivity and specific heat values exhibits much more scatter and irregularity. These results illustrate the importance of using test specimens that conform to the necessary criteria for a particular method.

The figure also contains results of measurements made up to approximately 380 °C using the Gustafsson probe. The results are in good agreement with the certified values except for the higher temperature measurements which are tending to diverge to higher values. The explanation appears to depend on the fact that a nickel sensor was used and since nickel undergoes a transition in the region of 350 °C this could influence the results obtained on the specimen. It should also be mentioned that while the standard deviation for the individual thermal conductivity values was never greater than 3.5%, that for the heat capacity and thermal diffusivity ranged from -4 to +10% indicating much more uncertainty in any derived values from these properties.

A further example relates also to the use of the Gustafsson probe on fluids and especially the influence of convection and contact resistance. Measurements have been undertaken at room temperature on a silicone oil and on water and agar gel using both 6.4 and 3.3 mm diameter probes. The collected results are shown in Figures 2a and 2b which also contain values for the oil obtained by guarded hot plate and transient hot wire methods.

For the silicone oil the effect of convection is quite apparent and although the effect is reduced by use of much smaller probe the effect is of the order of 10 to 12%. A similar effect is seen with water but not with the agar gel, which has essentially the same thermal conductivity as water, thus confirming the effect is due to convection.

Measurements were also undertaken on ice in two ways, first with the probe sandwiched tightly between two blocks of ice with a contact grease on the surfaces, and secondly by immersing the suspended probe in water and slowly freezing the system. The measured values were 1.79 and 2.33 W/m.K respectively. The latter value compares very well with the literature value of 2.38 W/m.K at the same temperature [3] and indicates that there was little or no effect of contact resistance at the surfaces.

Clearly any such differences and uncertainties create serious problems for the scientist and engineer requiring “reliable data” for whatever material or application of concern. One means towards resolution of the problem is the development of an acceptable test methodology.

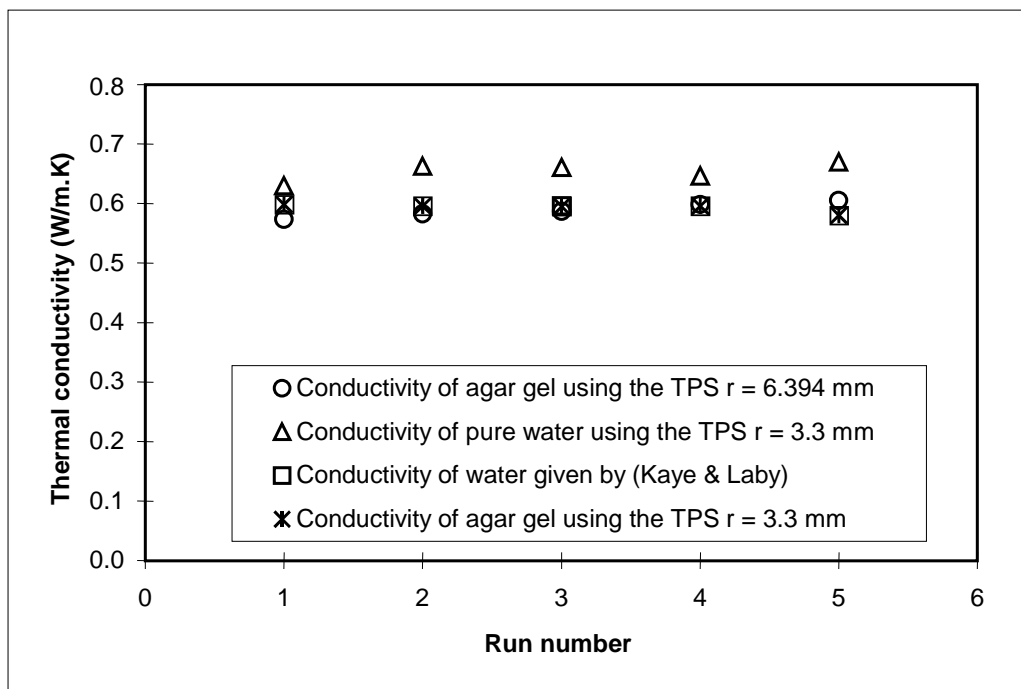


Figure 2a. Thermal conductivity of agar gel and water measured using transient plane source.

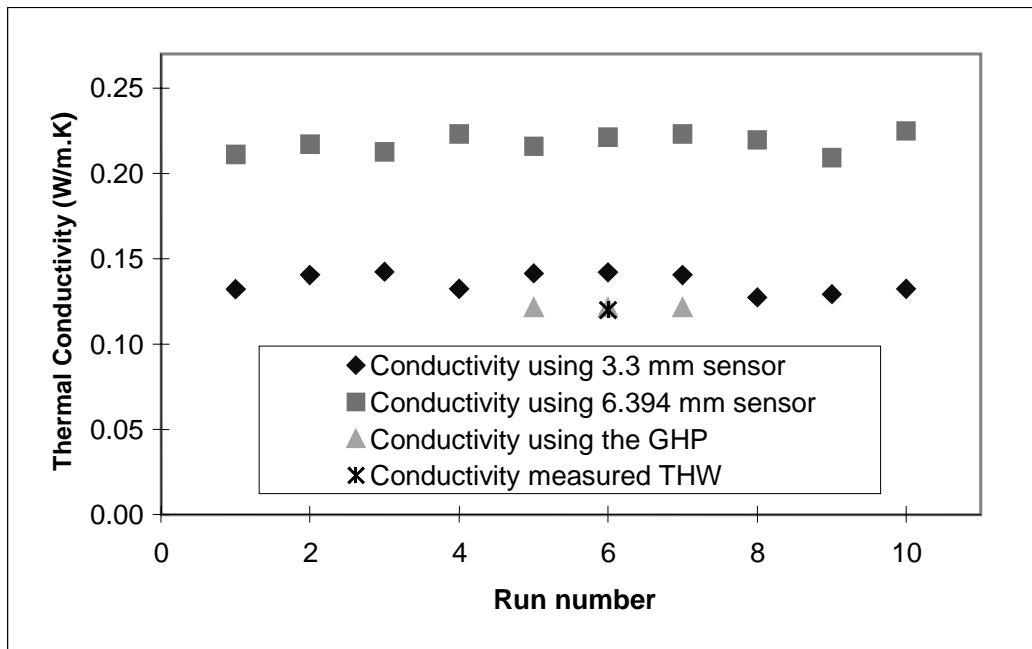


Figure 2b. Thermal conductivity of silicone oil by transient plane source, guarded hot plate and hot wire methods.

HISTORY OF DEVELOPMENT OF A STANDARD

Because of the worldwide use of these methods and the interests of so many workers, successive workshops addressing the subject have been held during the past three years in an attempt to resolve some of the issues and problems.

The first was organised by Ludovit Kubicar and held during the 14th European Conference on Thermophysical Properties (ECTP) in Wurzburg, Germany in October 1999. The second was coordinated by Ronald Tye and Ludovit Kubicar and held at the 26th International Thermal Conductivity Conference (ITCC) in Cambridge, Massachusetts in August 2001. The last was held in London at the 15th ECTP on September 2002. In each case some 40 to 50 attendees participated such that input was truly international.

Essentially the basic measurement procedure for these techniques is two fold:

1. Development of a model based on the common principle. This involves the solution of the partial differential equation for heat transfer in a specimen under selected or assumed initial and final boundary conditions.
2. Establishment of an experimental set-up designed to represent the particular model together with a set of solutions which describes the measurement process and develops the measured property(s).

To provide reliable data the experiment must represent the theoretical assumptions made in the development of the model. The measurement process involves the generation and subsequent mapping of a temperature field by a particular form of heat source (line, disc, strip etc.) and deriving the thermal properties from the experimental data using the model. Essentially the major source of any discrepancy(s) is the degree to which the experiment does not truly represent the model. Compounding this issue is the matter of the validity of the model being representative of the actual situation and potential effects of external parameters.

At the first meeting an attempt was made to present and discuss the basic issues represented by the above in order to:

- establish and prioritise the issues
- focus on means to address and resolve modelling and experimental issues
- formulate cooperation and coordination of effort

Although no overall solutions were obtained a number of participants did agree to share interests and cooperate on some of the issues in order to develop some form of common approach. It was generally accepted that this could only be obtained by a collective and cooperative international approach towards some form of standardisation.

The second meeting proved to be much more focused and covered the following:

- The variety of critical parameters that required consideration; in particular adequate specimen size and geometry, the power level and time interval of the heat pulse, the time window for data analysis, contact resistances (both external and internal) and sensitivity to boundary conditions.

- The need for commercial equipment suppliers to be more aware of the many issues involved and to provide means to address them and to issue better and more comprehensive instruction manuals, especially for equipment based on “black boxes” containing closed software over which the user had no control for undertaking internal checks.
- The problems arising when combined heat transmission mechanisms are involved and direct relationships between thermal properties may not exist.
- The need for additional reference materials and the requirements for known or certified values of λ , a and c .
- Which organisation or body national or international could or would be responsible for any standard(s) that may be produced.

The overall conclusions of the group were to:

- Continue initial objectives establishing an international network of experts.
- Produce a draft standard(s) covering measurement methods, using contact transient techniques and circulate amongst the key workers in the field for comment and amplification. NPL, with the cooperation of Ludovit Kubicar, agreed to accept this task.
- Identify candidate reference materials covering a broad range of thermal properties appropriate for use with transient techniques and develop a test protocol for comparison testing.
- Organise a further Workshop at the 16th ECTP in London in 2002 to present and discuss the draft standard and associated issues.

During the period between the second and third Workshops a first draft was prepared by the authors together with a number of inputs by experts involved in specific techniques. This was revised and refined and a second draft submitted for discussion at the meeting.

The document and its format as a generic main section combined with a number of annexes, each detailing a specific technique provided a source of much discussion and agreement. However

the final consensus was that the effort should proceed and revised documents based substantially on the draft prepared for general submission to the thermophysics community via the NPL website (<http://www.npl.co.uk/>) and efforts to be made to introduce the document into both the international and national standards communities.

THE CTM STANDARD

Essentially the document takes the form of a generic standard containing the basic information common to the techniques obtained in Table 1. This is supplemented with a series of normative annexes, each containing the additional detailed information specific to an individual technique, particularly the scope, influencing factors, apparatus and test specimen.

In general the overall format of the generic document and accompanying annexes is that of an international standard (ISO) while being similar to that of various national standards. Thus the contents consist of and Introduction, Scope, Referenced Documents, Terminology, Summary of Method, Significance and Use, Influencing Factored, Apparatus, Test Specimen, Procedure, Calculation of Properties, Verification of Method and Apparatus, Report, Precision and Bias and Bibliography. Brief details of each are provided but for reasons of space limitations only essential features of the generic document are presented. The annexes contain similar information that is specific to a particular technique.

(a) Scope

This states in broad detail the basic requirements of the family of methods that can provide one or more thermal properties obtained by analysis of the temperature /time response resulting from a heat pulse or heat flux in the form of a step wise function generated within a specimen by some form of simple heat source. Overall this family of techniques can cover the range of thermal conductivity $0.05 < \lambda < 200$ W/m.K, specific heat $200 < c < 2000$ J/kg.K and thermal diffusivity $0.01 \times 10^{-6} < a < 10 \times 10^{-6}$ m²/s in the temperature range $200 < T < 1600$ K. However reference is made to the annexes which contain the more limited ranges of properties and temperatures for each techniques and of the material types that can be investigated.

(b) Referenced Documents

This section contains a list of relevant international and national standards applicable to this methodology.

(c) Terminology

This is a complete list of terms and symbols that are used throughout the generic and annex documents.

(d) Summary of Method

This section describes the essential features of the test namely that an appropriately sized rectangular or cylindrical specimen containing an embedded simple geometric form of a low heat capacity heat source together with one or more combined or separate temperature sensors is allowed to equilibrate at a given temperature. An electrical current produces a heat pulse or heat flux in the form of a step-wise function in the electrical resistance (heat source) to generate a dynamic temperature field within the specimen. The temperature change with time (temperature response) is measured by a sensor(s), which is either unified with the heat source or placed a fixed distance from the source. The response is then analysed in accordance with a model and set of solutions (temperature functions) developed for the representative set-up and designed for the specific geometry and assumed boundary and initial conditions. Depending upon the geometry of the specimen and source and the means of the temperature field generation, one or more thermophysical properties can be obtained separately or simultaneously.

(e) Significance & Use

This outlines the potential benefit of the methods including:

- Suitability for broad ranges of materials, temperatures, conditions and environments
- Rapid assembly and measurement times coupled with relative simplicity of specimen configuration and measurement concept.
- Multiproperty in concept for essentially homogenous dense materials. However it is pointed out that this gain in number of properties can result in a corresponding loss of accuracy in one or more properties.
- A detailed inspection of a material structure is possible where one specimen is measured by several techniques involving a broad range of dynamics of the temperature field from a high level (pulse transient, followed by a low one (step-wise) to a low intensity using a small change hot-plate transient (Gustafsson disc).

(f) Influencing Factors

This section is a most important one since it draws attention to the major factors that influence the final precision of the various techniques and means to minimize the effects.

Two factors influence the accuracy of any transient method, namely the measuring time during which the temperature field is developed inside a specimen and the specimen and the heat source geometry as these limit the non-disturbed development of this temperature field. The optimal experimental set-up requires a specimen size such that the temperature field generated by the heat source will not be disturbed during the time period when the temperature response is highly sensitive to the thermophysical properties of the tested materials. The essential criterion for accuracy is to have a non-disturbed temperature field from that generated by the heat source. Two deviations occur when the real model is compared to the ideal one due to the finite size of the specimen and the structure of the heat source being different to the real one. The ideal heat source has negligible thickness, the heat source should be made of the same material and thermal contact between the specimen and heat source should be zero as shown in Figure 3. The ideal model gives an optional time of measurement and a maximum time window in which the evaluation can be made. This time window corresponds to the highest sensitivity coefficient where the correlation between them is minimal

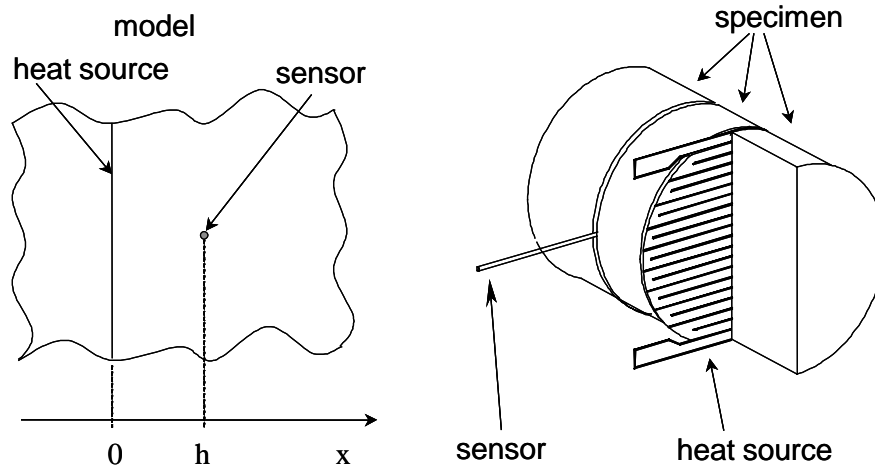


Figure 3. Difference between the ideal model and the real one for pulse transient and step-wise transient methods. A part of specimen is cut out to see the structure of the heat source.

From the methods shown in Table 1 three basic forms of temperature field exist as shown in Figure 4. It can be seen that three thermophysical parameters may be determined when a two probe system (source and temperature sensor) is used but that a one probe system will provide only one

unless a more complicated system is used. However the complicated symmetry puts high requirements on the control of the isotherm shapes.

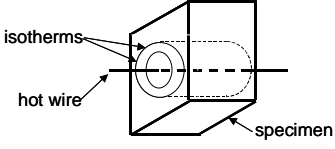
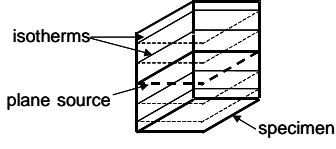
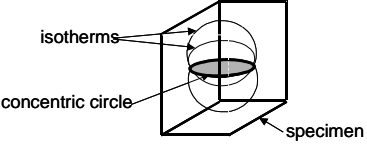
Line heat source	Plane heat source	Disc heat source
Hot wire Hot strip Needle probe	Pulse transient Step-wise transient Hot plate transient	Gustafsson probe
 <p>isotherms</p> <p>hot wire</p> <p>specimen</p> <p>Shape of isotherms: concentric cylinders</p>	 <p>isotherms</p> <p>plane source</p> <p>specimen</p> <p>Shape of isotherms: planes</p>	 <p>isotherms</p> <p>concentric circle</p> <p>specimen</p> <p>Shape of isotherms: ellipsoids</p>

Figure 4. Symmetries of the temperature fields given by the geometry of the heat source.

However the generated temperature field is distorted by the heat source, contact resistances and surface effects and thus there are deviations from the ideal model as illustrated in Figures 5-8. The experimental set-up has to be designed such that the volume corresponding to the deformed temperature field is negligible compared to that corresponding to the non-disturbed field. Generally the specimen volume corresponding to the deformed temperature field induced by thermal resistance is significantly larger in comparison to that caused by the construction resistance. Figure 9 is one example in which this difference is illustrated by an experiment run on Perspex. A difference analysis – as described in a later section – was used to obtain the time window as indicated by a period of data stability. The time window for the ideal model is limited by the sensitivity coefficient and by correlation the corresponding experimental time window is limited by the heat source effect at the beginning and the surface effect at the end.

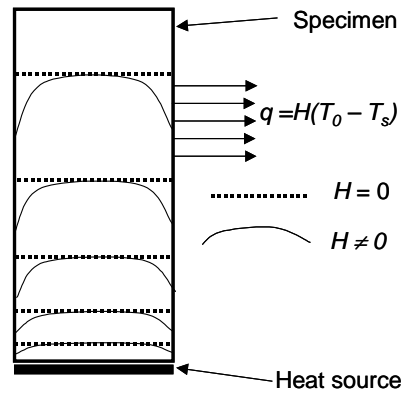


Figure 5. Deformation of the temperature field for plane heat source and the specimen in a form of cylinder. H – heat loss coefficient, T_s – specimen surface temperature, T_0 – surrounding temperature

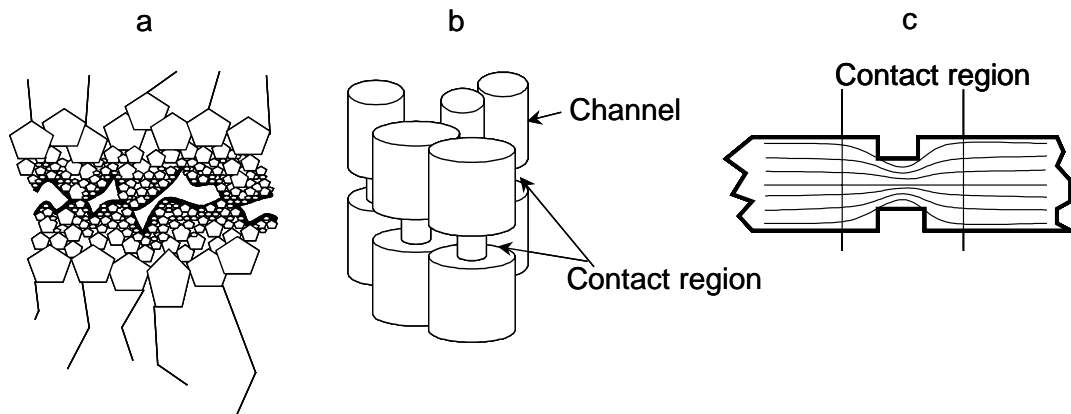


Figure 6. Constriction contact resistance. a – real contact of two bodies, b – idealized model showing a set of flux tubes connected by conducting spots where cross-section of tubes and contacting spots are the same as the cross-section of two contacting bodies and of the contacting surface points, respectively, c – contact region represents the tube volume corresponding to deformed flux lines (deformed temperature field)

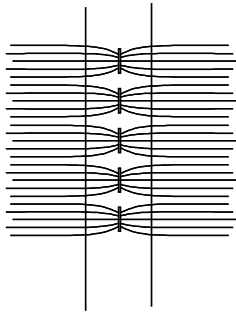


Figure 7. Induced constriction contact resistance due to structure of the heat source shown in Fig. 1.

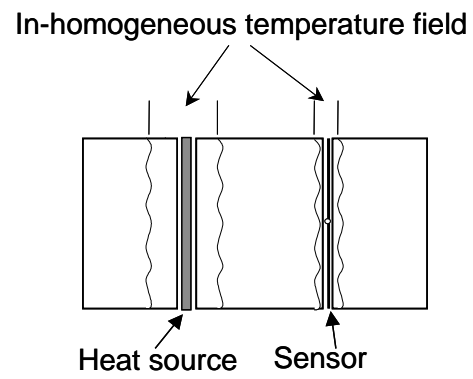


Figure 8. Deformation of the temperature field for heat source and for sensor regions

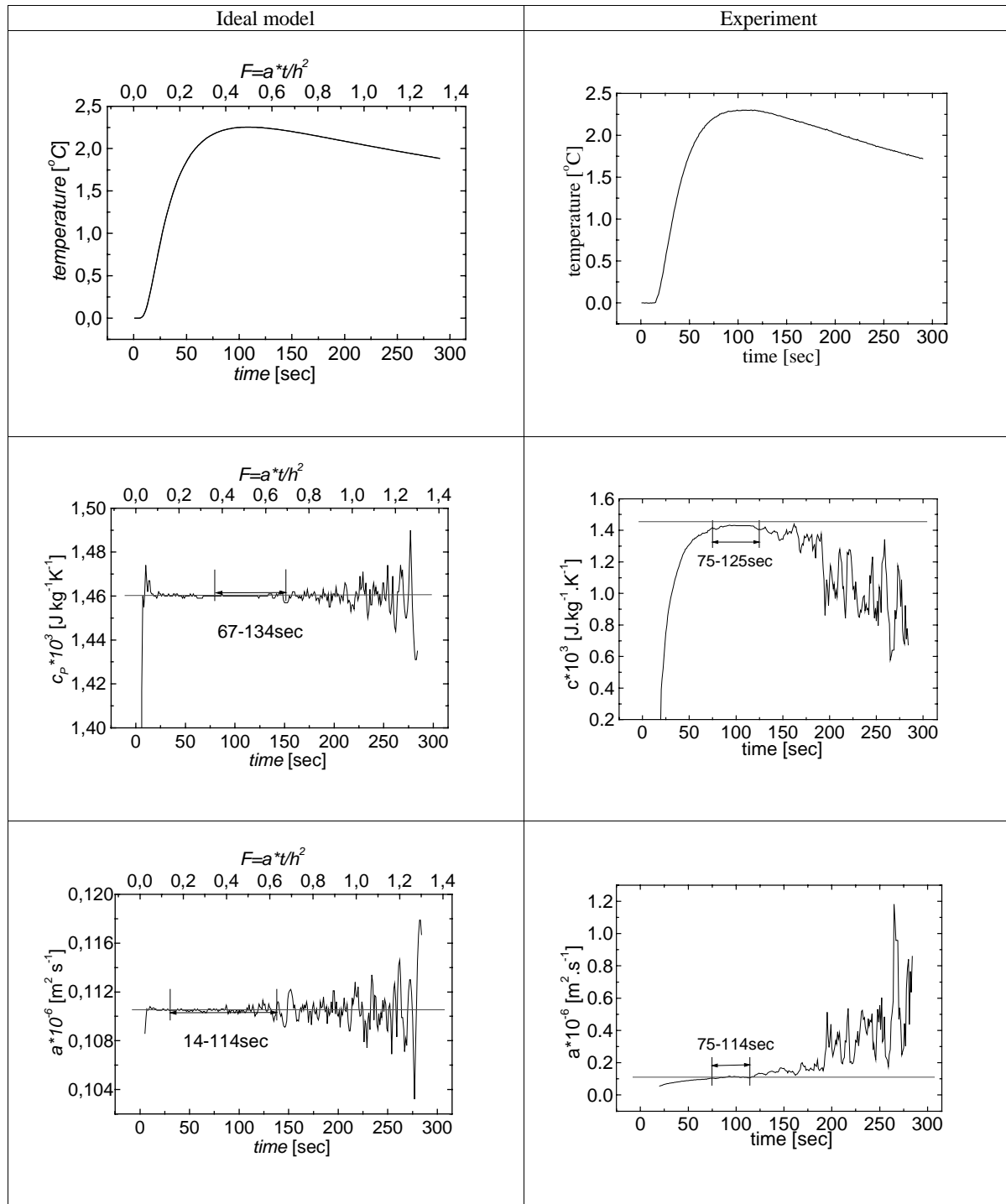


Figure 9. Difference analysis results for pulse transient method comparing the ideal model and experimental values (Perspex specimen $\lambda = 0.192$ W/m.K, $a = 0.11 \times 10^{-6}$ m²/s, $c = 1460$ J/kg.K and $\rho = 1184$ kg/m³)

Other factors which can also affect the result include power levels for differing ranges of thermal properties and specimen sizes, the heat pulse time interval and the heat flux in the form of a step-wise function by analysis of the temperature response. In addition there are effects of specimen sizes and configurations, anisotropy of properties and other heat transmission mechanisms that may be present. They include radiation, convection and mass transfer and can affect the validity of the basic assumption that all heat is transmitted by conduction.

(g) Apparatus

This section contains the essential details of the experimental set up and the criteria for the ideal modes of the various techniques. These are shown schematically in Figures 10 and 11 respectively.

Parameters described in detail include the various forms and configurations of the heat source and temperature sensors. Since it has been established that automated systems are to be used, recommended limits for times of test, recording times, frequency of data acquisition, limits of temperature rise and resolution of temperature are provided.

For data analysis a fully automated data collection, analysis and display system containing appropriate software is required to allow tests to be operated and the resultant transient curve analysed in accordance with the temperature functions developed from the model. The fitting procedure used should be applied over the whole temperature response in order to obtain a large enough time window for a reliable fit. This window is dependent on various parameters including specimen properties and size, power level and construction details of the power source and sensor. Means to evaluate optimum requirements are an essential requirement and shall be included in the software to allow internal consistency to be attained.

(h) Test Specimen

Overall the methods can be used for a broad range of materials of widely different properties but a particular method may be more appropriate for a particular type size and form of material and/or range of thermal properties. Thus a study of the model and its governing parameters is recommended to ensure that the most appropriate method is being used for the material to be tested.

It is particularly important to have a large enough specimen not only to ensure that it is suitable for the chosen method but also of sufficient size that the measured properties relate to the bulk material. As a minimum the active specimen volume should be at least 10 times greater than the characteristic size of any component or inhomogeneity and that surface effects should have zero effect on the measuring process.

Details are provided on containment of non-solid specimens, means to minimize contact resistances, means to evaluate anisotropic material and a standard conditioning procedure is recommended.

(i) Test Procedure

A detailed protocol is included to address the need for a reproducible test such that adequate comparison of data obtained by these methods can be made. This addresses density and form prior to and following a test, ensuring the specimen is correctly assembled and contact resistances minimised, stability of temperature prior to and following a test, point application of power input and recording of resultant temperature curve. Recommendations of number of repeat values ensuring stable temperatures prior to repeat runs especially in cases where change of physical state occurs. On completion of tests at the highest temperature level at least one repeat point is recommended on cooling.

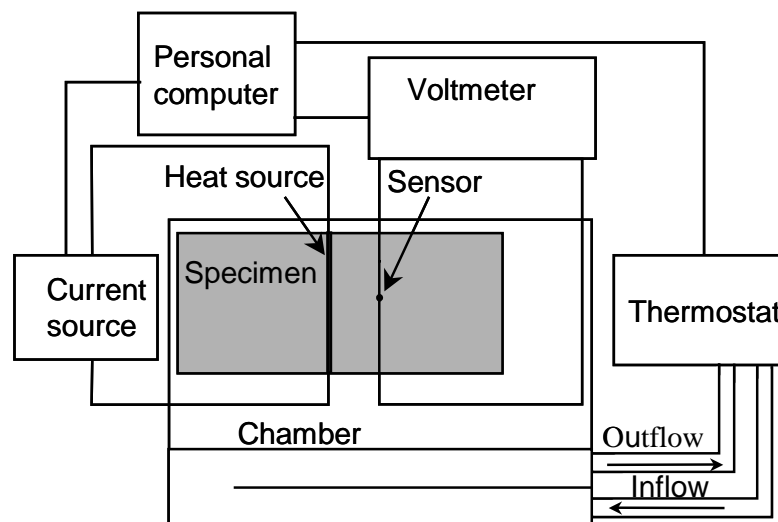
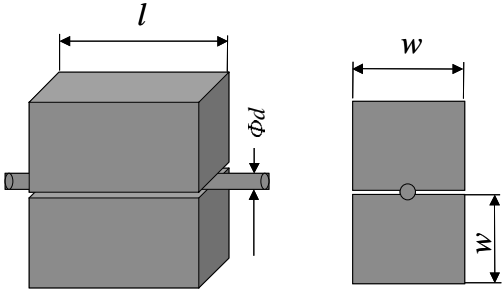
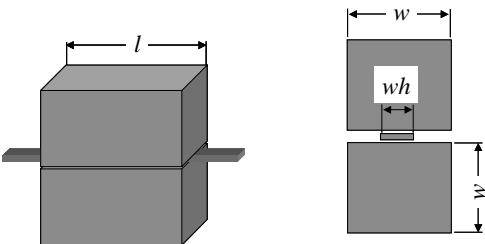
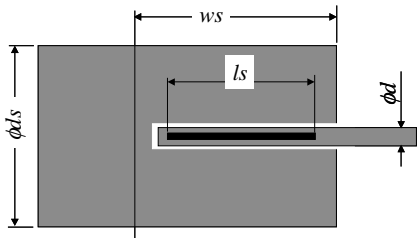
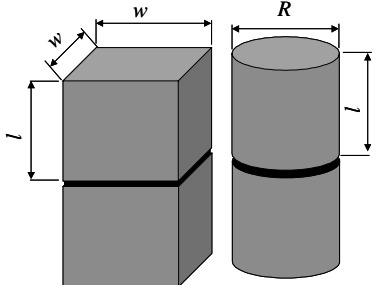
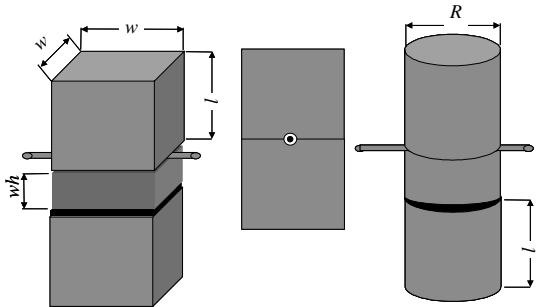


Figure 10. Block diagram of basic apparatus for transient methods.

Method	Specimen set up	Characteristic parameters	Criteria of ideal model
¹ Hot wire		l – length w – width Φd – wire diameter	$200d < w$ $l > 4w$
² Hot strip		l – length, broad w – width wh – strip width	$200wh < w$ $l > 4w$
¹ Needle probe		ws – active zone ls – needle probe length Φd – needle probe diameter Φs – specimen diameter	$ls > 100d$ $ws > 1.5ls$ $ds > 20 d$
³ Hot plate		w – width, broad l – height R - diameter	$w, l, R > 3\sqrt{at_{\max}}$ a – thermal diffusivity t_{\max} – maximal measuring time
³ Pulse and Step wise transient		w – width, broad l – height R – diameter wh – specimen thickness H – surface heat loss coefficient.	$l > 1. wh$ $R, w > 5wh$

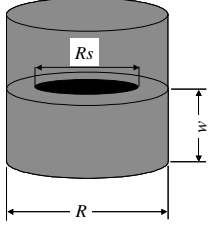
² Gustafsson probe		R_s – heat source diameter R – specimen diameter w – specimen thickness	$R_s - R > 5\sqrt{at_{max}}$ $w > 2.5\sqrt{at_{max}}$ $w > at_{max}$ t_{max} – maximal measuring time
-------------------------------	---	---	--

Figure 11. Schematic view of various experimental methods showing critical dimensions. The final column shows the criteria for the ideal modes of operation.

¹An appropriate groove has to be made for the hot-wire or needle probe. A good thermal contact has to be assured normally by the use of a heat sink paste otherwise the temperature field is disturbed.

²A good thermal contact has to be arranged between the strip or disc and the specimen parts. There should be no heat sink paste outside the heaters otherwise the temperature field can be disturbed. Enough uniaxial pressure should be applied to press specimen parts and the heater together.

(j) Calculation of Thermal Properties

Two basic forms of temperature response (temperature rise v time) are obtained depending on the methods used

Calculations of the thermophysical properties from the scans can be performed by fitting the corresponding temperature function in a chosen time window or from one point (it is the maximum of the temperature response) for the pulse transient method. Various calculation techniques are used to estimate the optimal time window as its choice is critical for data reliability. Depending on the model some of these curves can be re-plotted against the logarithm of time or square root of time to make the analysis easier.

While various approaches can be used a recommended approach is a difference analysis based on a fitting of the temperature response over a selected small time interval in which the fitting procedure is applied successfully over the whole temperature response in order to obtain a large enough time window for a good fit. The valid time window is indicated by constant values of the thermophysical parameters over a time range. After eliminating any early and later scans of the temperature response a time window as large as possible (middle period) should be obtained thereby ensuring that a large enough penetration depth has been attained. Figure 12 is an example of the schematic representation of the procedure for a specific method representing this technique. Where other calculation techniques are used providing their efficacy is demonstrated.

(k) Verification

While these methods may in general be described as being “absolute” in that every effort has been made to base the experiment and its operation in accordance with a model approximating the ideal one some uncertainties and inter-related effects may be present. Thus it is recommended that all apparatus be verified by utilizing reference materials. These can also be used to calibrate particular forms of a particular method which may be based on the use of a “known” specimen.

The standard contains data for currently available materials where certified or accepted property values are available. These are listed in Tables 2a to 2d. A strong plea is included for additional reference materials having all three properties known to acceptable limits to me made available.

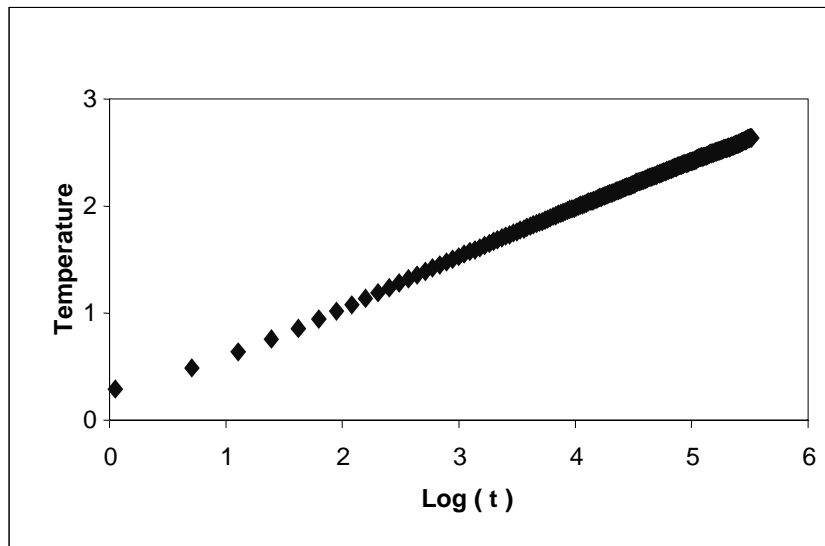


Figure 12a. Temperature response as a function of log time.

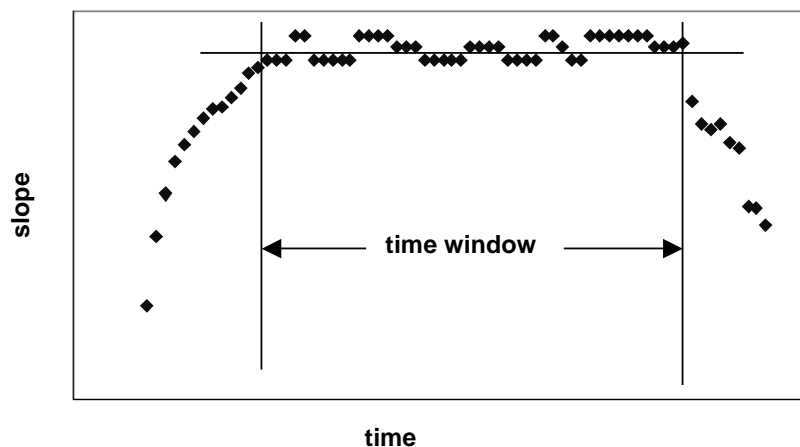


Figure 12b. Result of difference analysis of temperature response.

(l) Report

This is a standard list of basic requirements concerning the material(s) and specimen tested, how the test was undertaken, how the apparatus was verified, the property values and relevant details of experimental parameters used for the analysis.

(m) Precision and Bias

This has not yet been obtained. Currently and interlaboratory study is in process. This involves at least eight international organisations which use the Gustafsson probe. Several organisations which use one or more of the other techniques are also involved in order to evaluate the extent of agreement between techniques.

(n) Bibliography

This is a list of over 30 representative publications on the various techniques. The intent is that it will be continuously updated by interested parties.

SUMMARY

The present paper describes the need for, the historical development and contents of a standard method of test using contact transient methods of measurement which has been developed in accordance with the wishes and suggestions of workers in the thermophysical properties community. It is a continuing development and it is hoped that international and national standards bodies will benefit from its availability.

Table 2a. Thermal properties of polymethylmethacrylate (1180 to 1185 kg/m³) reference material

Temperature / °C	Thermal conductivity / (W/m·K)	Specific heat capacity / (J/g·K)
0	0.188	1.267
20	0.191	1.347
40	0.193	1.428
60	0.196	1.502
70	0.198	1.549

Table 2b. Thermal properties of Pyrex 7740 (2220 to 2225 kg/m³) reference material

Temperature / °C	Thermal conductivity / (W/m·K)	Specific heat capacity / (J/g·K)
0	1.10	0.720
50	1.18	0.810
100	1.24	0.878
200	1.33	0.991
300	1.45	1.082

Table 2c. Thermal properties of Pyroceram 9606 (2560 – 2600 kg/m³) reference material

Temperature / °C	Thermal conductivity / (W/m·K)	Specific heat capacity / (J/g·K)	Thermal diffusivity /(m ² /s)·10 ⁻⁶
25	4.06	0.821	1.93
50	3.92	0.851	1.77
100	3.71	0.902	1.60
200	3.42	0.982	1.36
300	3.23	1.038	1.23
400	3.10	1.079	1.14
500	3.00	1.110	1.07
600	2.92	1.135	1.02
700	2.86	1.156	0.972
800	2.81	1.177	0.938

Table 2d. Thermal properties of Pyroceram 9606 (2560 – 2600 kg/m³) reference material

Temperature / °C	Thermal conductivity / (W/m·K)	
	PDMS	Ottawa Sand (1640 kg/m ³)
20	0.160	0.28
40	0.156	0.29
60	0.152	
80	0.148	
100	0.144	

ACKNOWLEDGEMENTS

Special thanks to Ulf Hammerschmidt, Silas Gustafsson and Izaak van Haneghem for all their help and support.